NATIONAL BUREAU OF STANDARDS

# Technical Newsnor

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## NATIONAL BUREAU OF STANDARDS

# Technical News

BULLETIN

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#### March 1965

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COVER: NBS Standard Sample No. 711, a three-pound block of lead-silica glass. This glass viscosity standard, available from the NBS Office of Standard Reference Materials, will help improve the quality control of glass products (details on p. 43).

## Smog Attacks Roofing Asphalt

#### Study by J. R. Wright and P. G. Campbell, NBS Institute for Applied Technology

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Ozone, a common constituent of smog, is detrimental to roofing asphalt. The results of an NBS study <sup>1</sup> to determine the influence of environmental conditions on asphalt degradation show that the degradation rate depends upon such factors as the gaseous environment, the radiant-energy source, and the geographical source of the asphalt. When air is enriched with ozone, as is the case under smog conditions, the photodegradation rate of irradiated asphalt increases up to threefold.

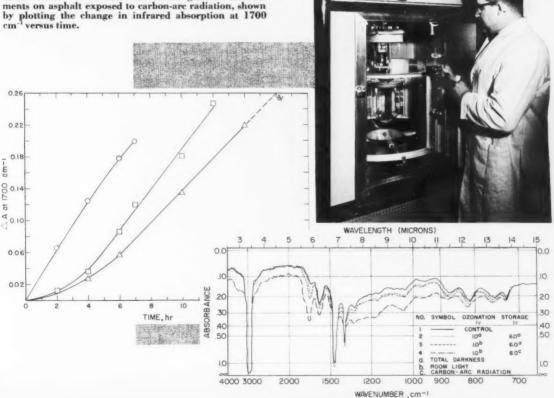
The problem of atmospheric smog has, in recent years, generated extensive research into atmospheric

oxidants other than oxygen and their reactions with organic materials in the presence of sunlight. Little attention, however, has been given to the reaction of asphalt, an organic material, to these oxidants, despite the fact that asphalt accounts for 80 percent of the roofing materials used in the United States today.

In this study four types of asphalts were prepared in thin-film specimens and subjected to three types of radiation in three different atmospheres. The asphalts were Southeastern U.S.A., mid-continent U.S.A., California Coastal, and California Coastal with a catalyst, FeCl<sub>3</sub>. The radiant energy sources and atmospheres, respectively, were carbon-arc, xenon-arc, and sunlight; and air, oxygen, and ozone-enriched oxygen.

Infrared spectroscopy was used to measure changes

Photo: P. G. Campbell inserts a specially made glass apparatus, containing an asphalt specimen within a selected atmosphere, into the carbon-arc weatherometer. Lower right: Infrared spectra of southeastern USA asphalt show the effects of ozone under various radiant-energy conditions. Below: The effects of various gaseous environments on asphalt exposed to carbon-arc radiation, shown by plotting the change in infrared absorption at 1700 cm<sup>-1</sup> versus time.





A 2-week, professional course in Electromagnetic Measurements and Standards will be offered August 9–20, 1965, by the Radio Standards Laboratory of the NBS Institute for Basic Standards, in association with the Bureau of Continuation Education of the University of Colorado. This course is intended for the professional staff within industry, university, military, and other government technical facilities whose responsibilities include precision measurements, quality control, standards, etc. Topics will include the theory of measurement and errors, review of basic electromagnetic theory, specification of the fundamental quantities of electromagnetic standards and their operational realization. Emphasis will be placed on the use of

standards to obtain the highest precision.

Prerequisites: Education equivalent to a B.S. degree in Electrical Engineering or Engineering Physics, and a year or more of actual working experience.

Tuition: \$200.00.

Registration Deadline: July 15, 1965.

Registration will be limited and early application should be made to ensure consideration. Registration will be closed July 15, 1965. Further details and registration forms are available from The Bureau of Continuation Education, University Memorial Center, Room 328, University of Colorado, Boulder, Colo., 80301.

#### Smog Attacks Roofing Asphalt (Cont.)

in the asphalts. An increase in absorbance in the carbonyl groups at 1700 cm<sup>-1</sup> indicates that either oxidation, photo-oxidation, or both have occurred. Infrared spectra of unexposed asphalts and those subjected to various exposure conditions were recorded. Comparisons of the spectra showed that all of the asphalts underwent changes in carbonyl absorbance under all conditions, except darkness. However, when exposed to ozone, three asphalts did exhibit changes in total darkness. The fourth, catalyzed California Coastal, did not react in the dark with ozone. Apparently the catalyst inhibited oxidation of the asphalt.

In photo-oxidation rate experiments, the duration of the induction period, the slope of the straight-line portion of the rate curve, and the time to film failure due to cracking were affected by the gaseous environment. The induction period in oxygen was less than that in air, but in ozone there was no induction period at all. When the slope for air is considered as unity, the ratio of the slopes for air, oxygen, and ozone-enriched oxygen are 1.0: 1: 1.27: 1; and 1.52: 1, respectively.

Overall photodegradation rates as measured by filmfailure time were approximately 18 hours for air, 12 hours for oxygen, and 7 hours for ozone-enriched oxygen. Thus, ozone caused an approximate threefold increase in photo-oxidation of the asphalts.

Although differences in the durability of the different asphalts to photo-oxidation were evident, the relative effects of air, oxygen, and ozone, and radiant energy sources were much the same. The California Coastal asphalt without FeCl<sub>3</sub> was more stable to photo-oxidation in ozone than was the catalyzed asphalt. This indicates that FeCl<sub>3</sub> may well accelerate photo-oxidation, yet inhibit oxidation in total darkness.

¹ Photo-oxidation of asphalts in the presence of ozone, by James R. Wright and Paul G. Campbell, J. Res. NBS 69C (Eng. and Instr.), (in press); Div. of Petroleum Chem. (ACS), preprints (Sept. 1964).



# Standard Materials

## **New Glass Viscosity Standard**

A second glass viscosity standard has been made available by the Office of Standard Reference Materials, NBS Institute for Materials Research. NBS Standard Sample No. 711, a homogeneous lead-silica glass, is for use in calibrating commercial glass viscometers. This standard, together with the first glass viscosity standard, NBS Standard Sample No. 710,1 will be helpful in improving the quality control of glass products. The viscosity-temperature relationship for this glass is similar to that of Standard Sample No. 710 but is displaced downward by approximately 100 °C.

Viscosity affects other physical properties of glass such as melting rate, devitrification temperature, crystallization characteristics, and the temperature and pressure ranges within which glass can be cast, pressed, drawn, or blown. Hence, to obtain glass products of uniform thickness, shape, and strength, particularly in high-speed mass production processes, the glass must conform closely to viscosity tolerances.

Institute staff members A. Napolitano and E. G. Hawkins made viscosity measurements on the lead-silica glass from 460 to 1360 °C. These data, together with data submitted by several research laboratories cooperating in the program, were tabulated in a certificate

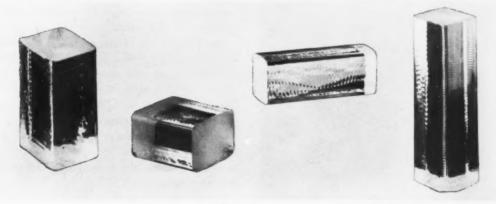
of viscosity values. The rotating cylinder method of measuring viscosities was used for temperatures above 700 °C where the glass is in a molten state. Below the softening point (approximately 602 °C), where the glass is in a plastic state, the fiber elongation method was used.

The softening, annealing, and strain points of the new standard glass have also been determined and are listed on the certificate. These measurements were made according to ASTM specifications.<sup>2</sup>

Standard Sample No. 711, a block approximately 2 in.×2 in.×6 in. and weighing about 3 lb, may be obtained from the Standard Samples Clerk, National Bureau of Standards, Washington, D.C., 20234 for \$60.00. A certificate of viscosity values is issued with each sample.

<sup>1</sup>Viscosity standard sample of glass, NBS Tech. News Bull. 46, No. 11, 174 (Nov. 1962); Viscosity of a standard soda-lime-silica glass, by A. Napolitano and E. G. Hawkins, J. Res. NBS 68A (Phys. and Chem.), No. 5, 439– 448 (Sept.—Oct. 1964).

<sup>2</sup> Softening point of glass, ASTM Standards, 1961; ASTM Designation: C338-57, pp. 695-698; Annealing point and strain point of glass, ASTM Standards, 1961, ASTM Designation C336-54T, pp. 645-650.



Sample No. 710, a soda-lime-silica glass (left) is a block approximately  $2 \times 3 \times 4$  in.; No. 711, a lead-silica glass (right) is a block about  $2 \times 2 \times 6$  in.

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## "Thumbtack" Accelerometer

Developed by P. Hertelendy and P. C. Reynard, NBS Institute for Basic Standards

Continuing research to improve instrumentation for physical measurements has led to the design of a miniature piezoelectric accelerometer, or vibration-sensing instrument.<sup>1</sup> This small accelerometer presents low mechanical impedance to the vibrating body and permits the precise location of measurement to be known.

Vibration measurements are becoming increasingly important in many areas of today's technology, providing a major area for NBS development work. Improvements in size, extension of the usable frequency range, and ease of fabrication are all needed. A small accelerometer was required in a recent project to monitor vibrations at certain points, for identification of vibrational nodes along the length of a metal bar. Also desired was a small transverse-to-axial response ratio so that, in effect, acceleration would be measured along



Pete Reynard positions a miniature accelerometer against a vibrating rod as Paul Hertelendy identifies vibrational node and peak indications on the oscilloscope. *Inset:* "Thumbtack" accelerometer can be seen between the vibrating system (bottom) and the positioning mechanism.

only one axis at a time. Such a ratio becomes increasingly important with increase in frequency; at the higher frequency ranges most vibrating surfaces have a three-dimensional motion and single-axis measurements can be made only by discriminating against transverse motions.

The vibration pickup devised for this application consists of a barium titanate piezoelectric disk sandwiched between a hardened steel probe and a cylindrical steel block. The steel block is the base by which the device is mounted. The probe consists of a 30° cone formed with a sensing tip of 0.25 mm radius at one end and at the other end a disk-shaped base 8 mm in diameter. As the probe looks much like a thumbtack, the device is called a "thumbtack" accelerometer.

The probe base is attached to a piezoelectric disk with conductive cement and the base block is similarly fastened to the other side of the disk. Electrical connection is made to the two sides of the piezoelectric disk through a ribbon lead welded to the block and another welded to the thumbtack base.

If the accelerometer base block is positioned with the probe facing the vibrating body and the tip in contact with it, the probe acts ideally as a spring and the more massive steel base as a seismic mass; the transducer responds primarily to motions along the axis of the device. A calibration in the range of 3 to 131 kHz showed the new accelerometer's median ratio of transverse-to-axial sensitivity to be 0.14, amply low for many experimental applications.

The thumbtack accelerometer offers the advantage of requiring only minute contact of the tip with the vibrating surface—less than 0.20 mm² with an aluminum vibrating surface that was tested, for example. This makes it easy to identify without ambiguity the point at which the vibration is being measured. Further, the compliant probe tip provides a low mechanical impedance to the vibrating body, reducing the extent to which the transducer distorts the vibration pattern in a compliant structure.

<sup>&</sup>lt;sup>1</sup> "Thumbtack" accelerometer for the 1.5-150 kc range, by P. Hertelendy and P. Reynard, Rev. Sci. Instr. 35, 1305-1306 (Oct. 1964).

## Calcium Carbide Method for Moisture Analysis Improved

The calcium carbide reaction method for the moisture analysis of solid materials has been improved in recent research.¹ Newly designed apparatus and test procedures provide determinations of moisture content for six specimens in less than 40 min. While not as definitive as a gas-chromatographic technique developed earlier at the Bureau,² the improved calcium carbide method should have numerous practical uses. It provides a simple, rapid means for the analysis of materials such as leather, paper, and grains, whose properties are influenced by their moisture content.

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In some procedures used for moisture analysis, the specimen is heated for stated intervals and the resulting weight loss is measured; however, water is sometimes reabsorbed in the process. In another technique, the specimen is mixed with calcium carbide and heated to drive out the water. The volume of acetylene generated by the reaction of the water with calcium carbide is then a measure of the moisture content of the specimen. The specificity and speed of this reaction should provide a sound basis for moisture analysis, but in exploratory tests at the Bureau poor precision was obtained.

Preliminary work to improve the method showed that when the calcium carbide reagent was heated, it produced variable gas volumes (blanks) that caused inaccurate test results. It was found that these variable blanks could be eliminated by sieving the reagent and heating it in a vacuum oven, with subsequent storage in a dry atmosphere until it was used.

A multiple-unit glass assembly was then designed, consisting of reaction flasks connected to calcium carbide flasks and to inverted burets each having a 35-ml capacity. Provisions for automatically heating and magnetically stirring the contents of the reaction flasks

were incorporated in the design as well as provisions for quickly bringing the assembly to initial thermal equilibrium at the conclusion of a test run. After this apparatus had been constructed, it was evaluated with several salt compounds having a known moisture content. A comparison of the known content with that found by reacting the compounds with calcium carbide in the apparatus showed excellent agreement.

To conduct a test, specimens of leather, paper, and grains were placed in reaction flasks. The burets were filled with mercury to the zero mark; the flasks containing calcium carbide were emptied into reaction flasks which were then immersed in a heating bath and stirred. Acetylene generated by the resulting reaction depressed the mercury by measured amounts in the burets. From the volume of acetylene, the moisture content of the specimen could be calculated with an overall experimental error of less than 2 percent.

Among the advantages of the method are the small specimen weights required, the rapidity with which results can be obtained as compared to other methods, the avoidance of moisture reabsorption during the process, and its general use for solid materials.

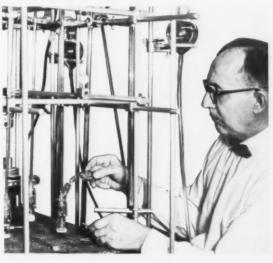
<sup>1</sup> For details, see Determination of moisture in solid materials by reaction with calcium carbide, by Sverre Dahl, Mater. Res. and Standards (in press).

<sup>2</sup> Gas chromatography in the determination of moisture in grain, by E. L. Weise, R. W. Burke, and J. K. Taylor, Humidity and Moisture Measurement and Control in Science and Industry, IV (In press, Reinhold Publ. Corp., New York, N.Y.); also, Determining moisture in grain by gas chromatography, NBS Tech. News Bull. 47, 116 (1963); and Problems in the determination of moisture, by J. K. Taylor, Report of the 48th National Conference on Weights and Measures 1963, NBS Misc. Publ. 254, pp. 70–76.

Left: One of the six glass assemblies used in moisture analysis. A, reaction flask; B, calcium carbide flask; C, buret; D, open mercury tube used to facilitate precise leveling with E, a mercury leveling bulb. Right: Sverre Dahl fits a calcium carbide flask to a reaction flask containing a ground leather specimen. Upper left and upper right are two mercury leveling bulbs.



Study by Sverre Dahl, NBS Institute for Materials Research



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## Thermal Emittage

Study by H. E. Clark and D. G. Moore, NBS Institute for Applied Technology, Under NASA Sponsorship

Most ceramic oxides have high melting temperatures and good chemical stability when heated in air. These properties make them useful in the space program for such applications as reentry nose cones and as linings for rocket motors and nozzles. However, for the ceramic oxides to be used with maximum advantage, their high-temperature thermophysical properties must be known.

One of the most important of these properties is thermal emittance. (Emittance is the ratio of the flux per unit area radiated by a specimen to that radiated by a blackbody at the same temperture and under the same conditions.) NBS scientists are currently working on equipment and procedures for obtaining reliable thermal emittance data for ceramic-type materials.

Although data exist in the literature on emittance of ceramic oxides, the published results vary over much wider limits than similar results for metals. These larger variations occur because reliable emittance measurements are more difficult to obtain for ceramics. Ceramics have very low electrical conductivity and cannot be heated by passing a current through them, as is done with metals. Also, as their thermal conductivity is low, ceramic specimens cannot be heated to a uniform temperature by placing them in contact with an electrically heated strip of metal. Finally, ceramics have relatively high transmission for radiation at certain wavelengths; thus, the energy emitted by a specimen can come not only from its surface, but also from appreciable depths below the surface. Therefore, if thermal gradients exist normal to the surface, it is impossible to assign a realistic temperature to the radiating material.

These problems were either eliminated or reduced to workable levels through use of a method in which specimens in the form of hollow cylinders are rotated at high speed in a furnace cavity with a water-cooled viewing port. With the present equipment, a given area on the specimen absorbs energy from the hot furnace walls for 90 percent of each revolution, and for 10 percent of the revolution it radiates through the viewing port. Because of the high rotational speed, thermal equilibrium is approached. Since gradients are largely eliminated by the rotation, a thermocouple placed in the specimen cavity indicates a temperature that closely approximates the temperature of the specimen surface.

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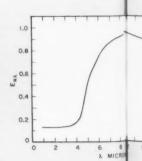
The energy per unit area per unit time radiated by the rotating specimen is compared directly to that radiated from a blackbody at the same temperature. An infrared spectrophotometer of the conventional double-beam ratio-recording type provides a monochromatic energy comparison at temperatures between 1200 and 1800  $^{\circ}$ K and for wavelengths of 1 to 15  $\mu$ . The resulting ratio is called the normal spectral emittance.

Measurements have been made on sintered specimens of aluminum oxide, magnesium oxide, and thorium oxide. The measured emittances in the 1 to 5  $\mu$  region were appreciably lower than literature values for similar materials. However, these low values were in good agreement with emittances predicted from reflectance measurements made on the same oxides. A thorough analysis of the method is in progress to increase the accuracy of the measurements.

Far right: Cutaway of specimen furnace used to achieve near thermal equilibrium in ceramic oxide specimens.

Below: Hypothetical curve showing how emittance of ceramic oxides at 1200 °K varies with wavelength.

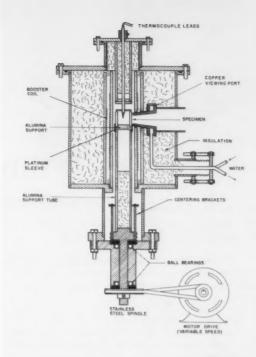
Photo: H. E. Clark adjusts current to one of the two blackbody furnaces shown (upper left) with the specimen furnace. Infrared spectrophotometer (right) is used to record emittance.



<sup>&</sup>lt;sup>1</sup> For further information, see Method and equipment for measuring thermal emittance of ceramic oxides from 120 to 1800 °K, by H. E. Clark and D. G. Moore, Symposium on the Thermal Radiation of Solids, NASA SP-55 (in press).

# tage of Ceramic Oxides

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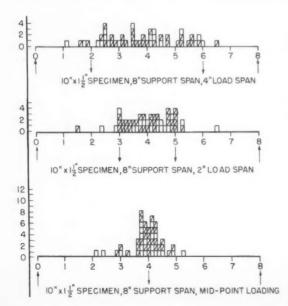
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# change recommended for trength Test of Flat Glass

Study by M. J. Kerper, T. G. Scuderi, and E. H. Eimer, **NBS** Institute for Materials Research

A change in the method of determining the strength of flat glass is recommended as a result of a recent study at the NBS Institute for Materials Research. Comparison of mid-point loading, recommended by ASTM, with two-point loading of flat glass has shown that the two-point loading tests give results that are more representative of the strength of the glass than those obtained from mid-point loading tests.1 The work was sponsored by the Air Force Aeronautical Systems Division.

In the NBS comparison tests, the ASTM standard method for determining the strength of flat glass 2 was followed except for variations in the specimen size and loading configuration. Eight separate tests were made; 50 specimens from each of two manufacturers were



used for all tests. When fracture occurred, the applied load was noted as well as the location and type (either surface or edge) of fracture.

The tests established that for two-point loading the glass will fracture at lower loads and the fractures will originate over a wider area of glass surface than in mid-point loading. In two-point loading tests, the fracture origins were evenly distributed in the area of uniform maximum stress between the loading knife edges with only a few fractures occurring outside this area. In mid-point loading, failures were concentrated around the point of load application where the stress is

The modulus of rupture, usually given to characterize the strength of flat glass, is the stress calculated as if the fracture occurred at the point of maximum bending moment. With single-point loading, the stress at the actual point of fracture may be significantly lower than the modulus of rupture. With two-point loading, the stress at the point of fracture and the modulus of rupture are identical as long as the fracture occurs at any point between the two loading knife edges. The fractures generally originate between the two loading knife edges in this type of test. Thus, the modulus of rupture-when based on two-point loading tests-is more representative of the practical strength of the

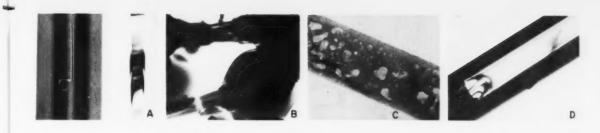
As two-point loading tests more closely approach expected loading conditions, it is recommended that the present mid-point loading test, as a flexure test for the strength of flat glass, be replaced with the following two-point loading test: a standard ASTM flat glass specimen (10 in. by 1.5 in. by 0.25 in.) supported over an 8-in. span and loaded with a 4-in. span.

For both mid-point and two-point loading, specimens in the same size group that failed from surface fractures tended to be stronger than those that failed from edge fractures. For this reason, it is recommended that in subsequent tests the type of fracture be reported for each specimen. Sufficient data would provide a separate criterion for evaluating the strength of flat glass.

Top of page: Recommended two-point loading test for determining the strength of flat glass: a standard ASTM flat glass specimen  $(10\times1.5\times0.25$  in.) supported over an 8-in. span and loaded with a 4-in. span. Left: Comparison of mid-point and two-point loading of flat glass, showing location of failures. Cross-hatched boxes represent surface failures, open boxes, edge failures.

<sup>&</sup>lt;sup>1</sup> Comparison of single-point loading and two-point loading for determining the strength of flat glass, by Matthew J. Kerper and Thomas G. Scuderi, Proc. ASTM (in press).

<sup>&</sup>lt;sup>2</sup> ASTM Standard Method C158-43.



## **ALUMINUM OXIDE WHISKERS**

## Studied by Electron Microscopy

#### Study by D. J. Barber, NBS Institute for Materials Research

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The thin, hairlike crystals called "whiskers" were once academic curiosities, but the discovery of their surprising strength has stimulated interest in their structure and properties. They are now being used in the semiconductor industry and their possible use as strengtheners in composite materials is being investigated.

A general study <sup>1</sup> of the morphology of aluminum oxide whiskers showed that whiskers grown by oxidizing molten aluminum globules fall into two classes: one, perfect crystals; the other, crystals with a dislocation (a line defect in the crystal lattice).

Whiskers were obtained by evaporating aluminum metal from the hot end of an evacuated aluminum oxide growth vessel and condensing it on the cooler walls at the other end; residual oxygen in the vacuum chamber oxidizes the highly reactive metal. This mode of crystal growth resulted in whiskers with a "drumstick" form, the stick of aluminum oxide terminating in a solidified globule of aluminum.

Many of the whiskers examined in an electron microscope at 100 kV were perfect parallel-sided ribbons, a few hundred nanometers in thickness. No disclocations exist in these ribbonlike whiskers. Heating a ribbon whisker in the electron beam produced crystal-lographic thermal etch-pits on the whisker face.

The second group of whiskers possessed approxi-

mately circular or hexagonal cross sections which were either solid or hollow. These tubular whiskers provide experimental evidence of the theory that axial dislocations in whiskers may be manifested as hollow cores.<sup>2</sup>

The aluminum globules terminating the whiskers often have a polygonal form. Moreover, when the globules are sufficiently thin to transmit the electron beam, the extinction fringes within them indicate they are single crystals. The polygonal appearance of the single crystal globules is a physical representation of the anisotropy of the surface free energy of the metal.

Strongly heating a metal globule in the electron microscope caused it to melt and "explode," spattering smaller globules onto the supporting carbon film. The molten globules striking the cool film immediately rose away from it on little stalks, forming new drumstick whiskers in the electron microscope. Apparently residual oxygen in the electron microscope vacuum oxidized the liquid metal droplets. This caused an oxide stem to grow at the point of contact between the globule and film, producing new drumstick whiskers. Other workers 3 have recently presented evidence for this mode of growth.

<sup>&</sup>lt;sup>1</sup> Study initiated at the Aluminium Laboratories Limited, Banbury, Oxford, England, and completed at the Bureau. For details, see Electron microscopy and diffraction of aluminium oxide whiskers, by D. J. Barber, Phil. Mag. 10, No. 103, 75 (July 1964).

<sup>&</sup>lt;sup>2</sup> F. C. Frank, Acta Cryst. 4, 497 (1951). <sup>3</sup> R. S. Wagner and W. C. Ellis, Appl. Phys. Letters 4, 89 (1964).

A, Two classes of whiskers: Perfect crystals (thin, ribbonlike whiskers, right) and those with axial dislocations (tubular or hollow core whiskers le/t).  $\times 14,200$ . B, Aluminum globules terminating whiskers often have a polygonal form.  $\times 66,000$ . C, Heating of ribbon whisker produced crystallographic thermal etch-pits on whisker faces.  $\times 20,700$ . D, Tip of broken tubular whisker shows the end of aluminum plug which partially filled the tube.  $\times 12,500$ .



#### Standards Conference Papers Available

A bound volume containing preprints of 52 papers dealing with both technical and management problems of standards laboratories was prepared for the October 1964 meetings in New York sponsored jointly by Instrument Society of America Measurements Standards Division (ISA MSD), the National Conference of Standards Laboratories (NCSL), and the Precision Measurements Association (PMA). Copies of this publication are still available and may be purchased from Instrument Society of America, 530 William Penn Place, Pittsburgh, Pennsylvania 15219. The cost is \$10 for members of the cooperating societies and \$15 for non-members; and the title is 19th Annual ISA Conference Proceedings-Vol. 19, 1964. Part I: Standards Laboratories and Measurement Standards. Preprints of the individual papers, including a few additional ones received too late for incorporation in the bound volume, are also available from the same source at \$0.50 for members and \$0.75 for nonmembers.

A summary of the topics discussed in these papers was given in the article "Plans for 1964 Standards Laboratory Conference: NCSL in Joint Meeting with ISA and PMA" which appeared on page 141 of the August 1964 issue of the *Technical News Bulletin*. Further details can be obtained from the ISA at the address given above.

#### New Calibration Service for Peak AC-DC Voltage Comparators

A calibration service for determining the accuracy of peak ac-dc voltage comparators at audio frequencies has been inaugurated by the NBS Institute for Basic Standards. A corresponding service has previously been available for rms-responding ac-dc transfer standards. Calibration of a transfer standard consists in measuring the ac-dc differences at significant points in the voltage-frequency domain of the device.<sup>1</sup>

An ac-dc comparator of proven accuracy is a basic standard in any laboratory where accurate a-c measurements are required. The new service will provide verification of the accuracy of the peak ac-dc ratio of peak comparators. Tests can be made at 50, 400,

1000, 2400, and 4800 Hz, at voltages of 10 to 20 V, and at fees twice those given in schedule 201.303. The overall accuracy is conservatively estimated at 0.01 percent. At present the service is offered only in the Electrical Instruments Section in Washington. However, if the demand warrants it, a similar service may be offered by the Electronic Calibration Center in Boulder. Methods for calibrating average-responding comparators and average-responding ac-dc converters are now being investigated.

A-c voltage measurements are based ultimately on the d-c standard cells which maintain the volt at the National Bureau of Standards. The transfer from direct to alternating voltage has in the past been made with various forms of rms ac-dc transfer comparators. These permit accuracies of 0.01 percent or better over wide voltage ranges at frequencies up to 20 kHz, even with waveforms containing several percent of harmonics. In fact, the rms value is usually the most meaningful one for distorted waveforms because it is affected much less by harmonics than peak or average values are and because it governs the interchange of electrical with other forms of energy.

However, there are occasions when the true peak value is desired. Recently, moreover, highly stable precalibrated a-c voltage sources with ultra-low distortion (less than 0.01 percent) have become available commercially, together with peak ac-dc comparators for calibrating them in terms of d-c standards. Though more limited in usefulness, the peak to d-c comparators are simpler than rms comparators in that they sample only one point of the voltage wave while rms comparators must correctly integrate the squared values along the entire wave.

#### R-F Voltmeters and Signal Sources: Higher Accuracies and Extended Range

The NBS Radio Standards Laboratory announces that the uncertainty of calibration of voltage measurement instruments used at high frequencies has been decreased from 0.1 to 0.05 percent in the voltage range from 0.2 to 50 volts. At present this improved accuracy in measurement is available at 30, 100, 300 kHz, 1 and 3 MHz.

For those instruments that measure voltages below 0.1 V the calibration range has been extended from

he former range of 100  $\mu$ V to 0.1 V to a range of 1  $\nu$ V to 0.1 V. The extended calibration service is available over a frequency range of 30 kHz to 900 MHz.

## Calibration of Capacitors at High Frequencies

Added Service: An extension to the three-terminal capacitance calibration service has been announced by the NBS Radio Standards Laboratory, Boulder, Colorado. Calibrations are now performed at 100 kHz, 465 kHz, and 1 MHz for fixed nominal values of capocitance of 10<sup>-2</sup>, 10<sup>-1</sup>, 10<sup>0</sup>, 10<sup>1</sup>, 10<sup>2</sup>, and 10<sup>3</sup> picofarads. High grade capacitors of these small values are useful as standards in the measurement of inter-electrode capacitance of vacuum tubes. This new service supplements that previously available at 465 kHz only—popular as an intermediate frequency (IF).

Discontinued Service: With improved rf connectors becoming available, many standards and instruments using the older type connectors are no longer appropriate for use as laboratory standards. This is particularly true of capacitors fitted with binding posts or bananaplug connectors. In a general effort to economize the use of precision coaxial connectors, the Bureau is discontinuing calibration services at frequencies above 100 kHz for all capacitors which utilize unshielded types of connectors. In those instances where a continuing requirement exists for such capacitors the user is referred to NBS Technical Note 201, A technique for extrapolating the 1 kHz values of secondary standards to high frequencies.

#### **Publications**

A number of documents are available from NBS on the topic of electrical measurements. One of these is a list of publications, LP 38, Electrical Units, Instruments, and Measurements, available without charge from the Publications Section, Room 500, South Building, National Bureau of Standards, Washington, D.C., 20234. This list is a selection of publications which describe the methods and equipment used in the establishment and maintenance of the electrical units and the standards that have been developed at NBS for the calibration of measuring apparatus. It includes references to older publications of the Bureau which describe basic principles and methods and are therefore still useful, but it does not include references to articles on electrical measurements at radio and higher frequencies. As many inquiries can best be answered by reference to a recognized national standard, or by reference to a particular textbook or handbook, a few such standards and books are also listed.

#### Color-Temperature Standards Revalued

Color appraisals of industrial materials and manufactured products are often made by visual or photoelectric inspection under a standardized light source. Such a source is frequently a tungsten-filament incan-

Changes required in assigned voltages, and implied changes in color temperatures for given color temperatures.

Adoption date	Color tem- perature	Required change in voltage	Implied change in color tem- perature
	(°K)	(Percent)	(°K)
May 1963 Oct. 1964	2000	+0.6	4
	2042	+0.6	4
	2200	+0.3	2
	2300	+0.2	2 2 2
	2353	+0.2	2
	2600	+0.6	6
	2854	+1.5	16
	2900	+1.5	16
	3000	+1.6	22
	3200	+1.9	24

descent lamp calibrated by the NBS Institute for Basic Standards.<sup>2</sup> Each year, dozens of these lamps are issued with an accompanying report listing each of the voltages required to make the lamps operate at specified color temperatures.<sup>3</sup>

In addition to their color-control applications by industry, the Bureau-calibrated incandescent lamps have other uses. For example, they may serve directly as color standards for incandescent lamps, or they may be employed to check the performance of photocells and photographic materials.

It is recognized that, with use, slight increases in the values assigned to the voltages may become necessary to make the lamps operate at the color temperatures given in the report. Hence, the standard lamps maintained by NBS for conducting lamp calibrations are periodically revalued by comparison with the national color-temperature scale.<sup>4</sup>

In the most recent evaluation completed in October 1964, voltage increases ranging from 0.2 to 1.9 percent over the color-temperature range from 2000 to 3200 °K were found necessary. The values for these adjustments are listed in the table. Similar changes should be made in the values assigned to the lamp standards of color temperature purchased just before the adoption dates of the new values shown in the table.

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<sup>&</sup>lt;sup>1</sup>See A-c voltage calibrations, NBS Tech. News Bull. 47, 132 (Aug. 1963).

<sup>&</sup>lt;sup>2</sup> For further information on these calibration services, see Federal Register 29, No. 246 (Dec. 18, 1964), or write the Photometry and Colorimetry Section, National Bureau of Standards, Washington, D.C. 20234.

<sup>&</sup>lt;sup>3</sup>The color temperature of a light source is the temperature of a blackbody radiator that has the same chromaticity as that of the source. Color temperature serves directly to define an incandescent lamp, and it serves indirectly to define other sources such as those for artificial daylight. These latter sources are produced by a combination of incandescent lamps with various blue filters.

<sup>&#</sup>x27;This scale, established in 1931, is based on three points of the absolute temperature scale. These points are realized by blackbody radiators at the freezing points of platinum (2042 °K), rhodium (2233 °K), and iridium (2716 °K).

# Publication Briefs

Note: Publications mentioned in this column, unless otherwise stated, are available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402, and through local U.S. Department of Commerce field offices. NBS Tech. Note 247, 40 cents; NBS Tech. Note 153, 30 cents; NBS Misc. Publ. 264, \$1.00.

#### NBS Technical Highlights Reports Research Developments

Progress in research programs designed to utilize science and technology in advancing economic growth is reported in 1964 Technical Highlights of the National Bureau of Standards.

During the period covered by 1964 Technical Highlights, NBS programs were regrouped into four institutes to permit closer identification with the specific needs of science, industry, and commerce. Under the Institute for Applied Technology were grouped NBS programs which directly stimulate technical innovation and industrial use of the results of modern science and technology. Responsibilities of this Institute include the dissemination of technical information to industry and the technical community, the development of performance criteria for certain technological products and services, the development of tools for analyzing largescale problems (e.g., transportation) that cut across Government-industry lines, and the analysis of problems associated with the development of new technology in industry.

Technical Highlights reviews these and other activities of the Bureau for fiscal year 1964. It describes projects involved in developing and maintaining the Nation's standards of physical measurement, and in developing basic data on the properties of matter and materials. It also gives the highlights of NBS work in radio propagation research, cryogenic (low-temperature) engineering, building research, and data processing. Technical Highlights is directed toward research and industrial management, as well as to engineers and scientists.

Typical reports in the 281-page Technical Highlights are on

—Establishment of a Clearinghouse for Federal Scientific and Technical Information within the NBS Institute for Applied Technology, through transfer of its predecessor, the Office of Technical Services.

—The discovery that the semiconductor strontium titanate becomes superconducting (completely loses its electrical resistance) at very low temperatures.

-Development of a new form of plasma—the brushcathode plasma—which is both stable and uniform and which thus lends itself to precision measurement of its characteristics.

—New research techniques for studying the excitation of atoms and molecules in energy ranges that occur in high-temperature plasmas.

—Establishment of an Office of Standard Reference Materials within NBS to provide a national focal point for work on standard materials used in industrial process control and acceptance testing.

—Development of a computer-type research apparatus for testing and recording the alertness of human subjects.

—An elastic weighing technique for on-site calibration of devices for measuring the thrust of missile and rocket engines.

—Development of a versatile, convenient-to-use instrument for studying laser frequencies.

—The work of the National Standard Reference Data System to provide a national storehouse of critically evaluated data in the physical sciences.

-Studies of odometer accuracy in rental cars and trucks.

—Designs which substantially improve the watercarrying characteristics of highway culverts.

—Significant advances in the theory of chain-folded polymer crystals.

Additional features include a report on the Bureau's broad cooperation with American industry and with Federal, state, and local government agencies. portant avenues of Bureau-industry cooperation include the NBS Office of Invention and Innovation, the NBS Textile and Apparel Technology Center, and the Research Associate Plan, conducted in collaboration with industrial and professional groups. The Bureau also works closely with national professional societies and standardization bodies, including the National Conference of Standards Laboratories. In matters dealing with international standards and the establishment of values for physical constants, the Bureau works through a large number of international groups, and frequently plays a major role in organizing international committees.

A complete list of the Bureau's publications for the fiscal year is also included in *Technical Highlights*.

#### Survey of Magnetic Thin Films Published

Magnetic materials deposited as thin films are the subject of much recent study and are now coming into use as the state-retaining material of some advanced computer memories. The growth of interest in these films is indicated by the increasing number of technical papers dealing with them. The Bureau has published Technical Note 247 to give physicists and engineers studying or using thin magnetic films a survey of the literature and data on them.

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The new technical note, a 55-page publication entitled "Survey of Magnetic Thin Film Materials," was written by George Reimherr of the NBS Institute for Applied Technology, Information Technology Division. It is the second survey produced by this author, the first being Technical Note 153, "A General Survey of the Semiconductor Field."

Parameters of magnetization for 33 structures of 23 materials are given by the new publication in table format, supplemented by textual comments. Another table gives interesting features and potential applications for the materials. The source of each datum in a 201-reference bibliography is identified and an index facilitates finding all references to each material listed.

#### New Book on Liquid Hydrogen

R. B. Scott, Manager of the NBS Boulder (Colo.) Laboratories, is a co-editor of a new book, Technology and Uses of Liquid Hydrogen (Pergamon Press, Oxford, England), along with W. H. Denton and C. M. Nichols of the Atomic Energy Research Establishment, Harwell, England. The book is a compilation of advanced technical information from the foremost laboratories in the United States, England, Switzerland, and West Germany on the production and uses of liquid hydrogen. It covers the technical aspects of the production of hydrogen gas for liquefaction, hydrogen liquefiers, insulation requirements for storage and transport, safety practices, properties of hydrogen, and uses ranging from rocket fuel to refrigerant.

In his introduction Mr. Scott sketches the history of liquid hydrogen from 1898, when Sir James Dewar first liquefied it, to the present, and anticipates future developments. Some of the developments he foresees are the use of deuterium (heavy hydrogen) in nuclear fusion to provide the world with unlimited power, and the "taming of the hydrogen bomb" for peaceful purposes to benefit all mankind.

In addition to Mr. Scott, Bureau staff members B. W. Birmingham, D. B. Chelton, R. B. Stewart, and Hans M. Roder are authors and coauthors of several chapters.

### **Publications of the National Bureau of Standards**

#### Periodicals

Technical News Bulletin, Vol. 49, No. 2, February 1965. 15 cents. Annual subscription: \$1.50; 75 cents additional for foreign mailing. Available on a 1-, 2-, or 3-year subscription

CRPL Ionospheric Predictions for May 1965. Three months in advance. Number 26, issued February 1965. 15 cents. Annual subscription: \$2.50; 75 cents additional for foreign mail-

ing. Available on a 1-, 2-, or 3-year subscription basis.

Journal of Research of the National Bureau of Standards

Section A. Physics and Chemistry. Issued six times a year.

Annual subscription: Domestic, \$4; foreign, \$4.75; single copy, 70 cents.

Section B. Mathematics and Mathematical Physics. Issued quarterly. Annual subscription: Domestic, \$2.25; foreign, \$2.75; single copy, 75 cents.

Section C. Engineering and Instrumentation. Issued quar-Annual subscription: Domestic, \$2.25; foreign, \$2.75; single copy, 75 cents.

Section D. Radio Science. Issued monthly. Annual sub-

scription: Domestic, \$9; foreign, \$11.50; single copy, \$1.00.

#### Current Issues of the Journal of Research

R. W. Stanley.

Phase equilibria in the system vanadium oxide-niobium oxide.

J. L. Waring and R. S. Roth.

Thermodynamics of the ternary system: water glycine-potassium chloride at 25 °C from vapor pressure measurements. V. E. Bower and R. A. Robinson.

Heat of formation of aluminum fluoride by direct combination of the elements. E. S. Domalski and G. T. Armstrong.

Relative enthalpy of polytetrafluoroethylene from 0 to 440 °C. T. B. Douglas and A. W. Harman.

Anionic polymerization of isoprene at low concentrations of polyisoprenyllithium. L. J. Fetters.

One-particle transitions and correlation in quantum mechanics. A. R. Ruffa.

Disaccommodation of magnetic spectra of two manganese zinc ferrites. A. L. Rasmussen.

Splitting of a set of equivalent sites in centrosymmetric space groups into subsets under homogeneous stress. J. B. Wachtman, Jr., and H. S. Peiser.

Radio Sci. J. Res. NBS/URSI 69D, No. 3 (Mar. 1965).

Propagation in nonuniform gyrotropic media. S. H. Gross

and L. B. Felsen. Geometrical optics for gyrotropic bodies. W. C. Y. Lee, L. Peters, Jr., and C. H. Walter.

Attenuation of hydromagnetic waves in the ionosphere. S.-I. Akasofu.

Self distortion of radio signals in the D region. L. R. Megill. Atmospheric gravity waves: a new toy for the wave theorist. C. O. Hines.

Electromagnetic fluctuations in an equilibrium plasma. R. E.

A generalized hydromagetic wave in an inhomogeneous, cylindrical plasma. C. K. McLane and T. Tsukishima.

Angular dependence of the refractive index in the ionosphere. G. A. Deschamps.

Electrodynamics of moving anisotropic media: the first-order theory. C. T. Tai.

Study of the phenomenon of whistler echoes. T. Laaspere, W. C. Johnson, and J. F. Walkup.

Multiple-frequency investigations of radio wave absorption during the dawn-breakup phase of auroras. R. Parthasarathy and F. T. Berkey.

Sferic excitation of a two-layer conducting medium. M. B. Kraichman.

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DIVISION OF PUBLIC DOCUMENTS WASHINGTON, D.C. 20402

OFFICIAL BUSINESS

Currents, charges, and near fields of cylindrical antennas. R. W. P. King and T. T. Wu.

A note on the radiation conductance of an axial slot on a cylinder. C. M. Knop and C. T. Swift.

Analytical formulas for radio paths in spherically stratified ionospheres. E. Woyk (Chvojkova).

#### Other NBS Publications

On the statistical theory of electromagnetic waves in a fluc-tuating medium (II). Mathematical basis of the analogies to quantum field theory, K. Furutsu, NBS Mono. 79 (Dec. 7, 1964), 35 cents.

Investigation of the hydraulics of horizontal drains in plumbing systems, R. S. Wyly, NBS Mono, 86 (Dec. 18, 1964), 35

Aluminum nails, CS263-64 (Nov. 1, 1964), 10 cents

On the pictorial structure of Chinese characters, B. K. Rankin, III, W. A. Sillars, and R. W. Hsu, NBS Tech. Note 254 (Jan. 4, 1965), 30 cents.

Characteristics of the earth-ionosphere waveguide for VLF radio waves, J. R. Wait and K. P. Spies, NBS Tech. Note 300 (Dec.

30, 1964), 50 cents.

On the formulation and numerical evaluation of a set of twophrase flown equations modeling the cooldown process, S. Jarvis, Jr., NBS Tech. Note 301 (Jan. 1965), 55 cents.

National standard reference data system, plan of operation, E. L. Brady and M. B. Wallenstein, NSRDS-NBS1 (Dec. 30, 1964), 15 cents.

#### Publications in Other Journals

This column lists all publications by the NBS staff, as soon after issuance as practical. For completeness, earlier references not previously reported may be included from time to time.

Effect of electrical fields in the gamma radiolysis of propane, P. Ausloos and R. Gorden, Jr., J. Chem. Phys. 41, No. 5, 1278-1284 (Sept. 1, 1964).

Mass spectrometric investigation of the yttrium chlorine surface reaction, J. D. McKinley, J. Chem. Phys. 41, No. 9, 2814-2817 (Nov. 1, 1964).

Angular momentum distribution and emission spectrum of OH Σ+) in the photodissociation of H2O, T. Carrington, J. Chem. Phys. 41, No. 7, 2012-2018 (Oct. 1, 1964)

Kinetics of the acid-catalyzed hydrolysis of acetal in dimethyl sulfoxide-water solvents at 15, 25, and 35°, R. K. Wolford, J. Phys. Chem. **68**, No. 11, 3392-3398 (1964).

Morphology of thermally evaporated zinc cleavage surfaces, A. . Ruff, Jr., J. Chem. Phys. 41, No. 5, 1204-1213 (Sept. 1,

Simple arc devices for spectral excitation in controlled atmospheres, M. Margoshes and B. F. Scribner, J. Appl. Spectry. 18, No. 5, 154-155 (1964).

Collision-induced microwave absorption in compressed gases. III. CO2-foreign-gas mixtures, A. A. Maryott and S. J. Kryder, J. Chem. Phys. 41, No. 6, 1580-1582 (Sept. 1964).

Making precision measurements of zener diode voltages, W. G. Eicke, Jr., IEEE Trans. Commun. Elec. CE-83, No. 74, 433-438 (Sept. 1964).

Ethylene-propylene copolymers: crystallinity, infrared and creep studies, F. J. Linnig, E. J. Parks, and L. A. Wood, J. Appl. Polymer Sci. 8, 2645-2651 (1964).

Statistical surface thermoydynamics of simple liquid mixtures, C. A. Eckert and J. M. Prausnitz, A.I.C.h.E. J. 10, No. 5, 677-683 (Sept. 1964).

The accurate measurement of voltage ratios of inductive voltage dividers, Acta IMEKO, pp. 317-331 (1964)

The National Bureau of Standards, E. L. Graminski, Capital Chemist 14, No. 9, 2778-2781 (Dec. 1964).

Correlation factors for impurity diffusion-Bcc, diamond, and Fcc structures, J. R. Manning, Phys. Rev. 136, No. 6A, A1758-A1766 (Dec. 14, 1964).

Thermal decomposition of some alkyl halides by a shock-tube method, W. Tsang, J. Chem. Phys. 41, No. 8, 2487-2494 (Oct. 15, 1964).

Relaxation of a Lorentz gas with repulsive r-8 force law, H. Oser, K. E. Shuler, and G. H. Weiss, J. Chem. Phys. 41, No. 9, 2661-2666 (Nov. 1, 1964).

Sampling and statistical design, W. J. Youden (Proc. Symp. Environmental Measurements, Cincinnati, Ohio, Sept. 4, 1963), Public Health Service No. 999-AP-15, pp. 35-39 (July 1964)

Plastics and the dental market, G. M. Brauer, Plastics World 12, 12 (Oct. 1964).

NMR studies of asymmetric ethanic rotators: 1,2-disubstituted propanes, H. Finegold, J. Chem. Phys. 41, No. 6, 1808-1818 (Sept. 15, 1964).

Triplet-state energy transfer from acetone to aliphatic aldehydes in the gas phase, R. E. Rebbert and P. Ausloos, J. Am. Chem.

Soc. 86, 4803-4807 (1964).

The needs of the statistical engineering programs of the National Bureau of Standards, C. Eisenhart, Proc. 9th Conf. Design of Experiments in Army Research Development and Testing, Directorate of Research and Development, U.S. Army Missile Command, Redstone Arsenal, Ala., Oct. 23-25, 1963, ARO-D Report 64-2 (1964).

Impact parameter treatment of vibrational excitation, F. H. Mies,

J. Chem. Phys. 41, No. 3, 903–904 (Aug. 1, 1964).
The inverse multiplier for Abelian group difference sets, E. C. Johnsen, Can. J. Math. 16, 787–796 (Sept. 4, 1964).

Studies on the tungsten-rhenium thermocouple to 2000 °C, D. B. Thomas, Proc. 18th Annual ISA Conf. and Exhibit, Sept. 9-12, 1963, Chicago, Ill., Preprint 57.3.63, pp. 57.3.63-1-57.3.63-9 (1963).

Publications for which a price is indicated are available by purchase from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402 (foreign postage, one-fourth additional). Reprints from outside journals and the NBS Journal of Research may often be obtained directly from the authors.

#### Patents Granted on NBS Inventions

The following U.S. patents have recently been granted on NBS inventions and, except as noted, are assigned to the United States of America as represented by the Secretary of Commerce.

3,150,163 150,163 September 22, 1964. Pentafluorobenzonitrile and method of making the same. Walter J. Pummer and Leo A. Wall (Navv).

November 10, 1964. High-density, nonmagnetic 3,156,558 stainless steel. Samuel J. Rosenberg and Thomas P. Royston, Jr.

3,158,825 November 24, 1964. Movable resonant cavity tuning probe in dielectric sleeve having nonuniform outer Maurice J. Veter. surface.

December 8, 1964. 3,160,764 Pulse shaper employing charge-storing semiconductor device. George G. Harman, Jr., and Richard L. Raybold.

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